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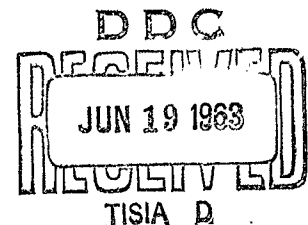
Studies of Solids at Low Temperatures

between

The Air Force Office of Scientific Research

and

The University of Alabama



Submitted April 8, 1963

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## 1. Electron Spin Resonance of X-Irradiated $\text{KH}_2\text{PO}_4$ and $\text{KD}_2\text{PO}_4$

### (a) Introduction

While considerable work has been carried out on the identification of free radicals produced in single crystals by x-irradiation at room temperature, relatively little work has been done on radicals which are only stable at low temperatures. The purpose of this research is to investigate radical species which are stable only at low temperatures by electron spin resonance and optical methods and to later study the kinetics of free radical formation and decay.

The initial compound chosen for study was  $\text{KH}_2\text{PO}_4$ , the deuterated compound also being included in this investigation. 50 KV x-rays produce free radicals in this compound which appear to be completely stable at liquid air temperatures, but decay rapidly above about 140°K. Complete orientation data have been taken on this compound about all three crystallographic axes for both undeuterated and deuterated crystals. The spectra can be interpreted as an isotropic hyperfine coupling to the  $^{31}\text{P}$  nucleus with about 1% 3s spin density on the phosphorus and the rest in a P orbital on the oxygen. The g tensor is highly anisotropic and exhibits, as would be expected, the symmetry of the crystal. Identical spectra are observed for the deuterated crystal except the lines are narrower by about a factor of 2 and two weak lines are observed which have the same isotropic hyperfine coupling but no g anisotropy. The model predicted on this basis is a  $\text{HO}_3\text{PO}\cdot$  radical.

(b) Instrumentation

The spectrometer is an x-band bridge type utilizing bolometer detection. The output is fed into a lock-in amplifier which records the signal derivative on an Esterline-Angus chart recorder. The magnetic field is modulated from the signal generator contained in the lock-in amplifier, and since we employ bolometer detection our modulating frequencies are of the order of 300 cps.

The microwave power was at one time furnished by a Varian V-58 klystron which was stabilized to the sample cavity. The entire stabilizer was constructed in this laboratory, and it proved satisfactory for room temperature measurements and all other cases where moderate amounts of power could be fed into the sample cavity. Our constructed stabilizer proved difficult to operate at very low power levels and we now utilize a Strand Labs, Inc. Model 500, external cavity stabilizer. This stabilizer also employs a Varian V-58 tube.

Two different sample cavities are now in use; a rectangular, metal, cavity operating in the  $TE_{011}$  mode, and an identical cavity constructed from Grade A Lava. (American Lava Corporation, Chattanooga, Tennessee.) The ceramic cavity was chemically silver plated and then a thin plate of gold was electrodeposited over the silver. Each cavity has an unloaded  $Q$  of about 5000. The lava cavity is used at helium temperatures, and the metal cavity is used at all other temperatures.

A Harvey-Wells magnet with rotating base and power supply is employed. The gap is 2.50 inches. The magnet has 12 inches diameter pole caps. A custom built helium dewar constructed by Superior Air Products is mounted

vertically in the field, and the magnet is capable of 180 degree rotation about this point. The dewar is all stainless steel with about a one liter helium capacity. In operation, the helium will last from 6 to 8 hours. The first three models of this dewar turned out to be defective, but after Superior Air Products Company replaced the inner dewar it has been satisfactory in all respects.

The x-irradiation damage to single crystals is carried out on a Phillips x-ray diffraction unit with a high current tube which is capable of giving 50 KV at 40 ma. We also have a diffraction unit with both tungsten and copper target tubes for alignment of the crystals.

The most recent work has been with potassium phosphate (Dihydrogen),  $\text{KH}_2\text{PO}_4$ . Single crystals of this compound were grown from aqueous solution. The crystals were in all cases suspended in the solution by a thread. They were removed when they were about 3 mm x 3 mm x 10 mm in size. Back reflection Laue patterns were run on these crystals to determine the crystallographic axis. Single crystals of  $\text{KD}_2\text{PO}_4$  were also grown in the same manner as above. The potassium dideuterophosphate was obtained from Merck Sharp and Dohme of Canada Ltd., and deuterated crystals were grown in  $\text{D}_2\text{O}$ .

#### (c) Results

The  $\text{KH}_2\text{PO}_4$  structure belongs to the tetragonal symmetry class  $D_{2d}^{12} - \bar{4}2d$ , with four molecules per unit cell. Some molecules are rotated about  $30^\circ$  in the ab-plane compared with other molecules.

A crystal was immersed in liquid air and irradiated overnight at 50 KV and 35 ma. It turned a deep red color. The crystal was maintained at

liquid air temperature for the duration of the experiment. The 2-fold rotational spectrum about the c-axis consists of eight lines in general; four lines where the external field is directed along the a- or b-axis, and four lines when the field is at  $45^\circ$  to these axes. The eight lines are thought to be 4 doublets of constant (33 gauss) separation arising from a spin on an oxygen interacting with a  $^{31}\text{P}$  nucleus. These doublets show a large anisotropy in g. Rotations about each of the other 2-fold axes show the same spectrum for each rotation. This spectrum also consists of 8 lines in general, with the simplest being a single doublet occurring when the magnetic field lies along the c-axis. The separation of the doublet is again about 33 gauss. The orientation dependence of the spectrum is shown in Fig. 1 and 2. At this point,  $\text{KD}_2\text{PO}_4$  crystals were grown and irradiated in the same way as the  $\text{KH}_2\text{PO}_4$  crystals. The esr spectrum about all axes is, within experimental error, the same as that of  $\text{KH}_2\text{PO}_4$ . Separations of 33 gauss are still measured and the same anisotropy in g is still observed. We did note that the deuterated crystals color faster under x-ray bombardment than do the  $\text{KH}_2\text{PO}_4$  crystals. The linewidth in the deuterated crystals is half that observed in  $\text{KH}_2\text{PO}_4$  crystals. Also two weak lines appear in the deuterated case which are not present in the undeuterated case. These lines have approximately the same splitting (29 gauss) as the other doublets but show very little or no g anisotropy.

The g tensor and the principal g values are, as calculated from the deuteron data by the method outlined by Schonland<sup>3</sup> are



$$\begin{pmatrix} 4.0273 & -.0309 & -.0215 \\ -.0309 & 4.1205 & \pm .0708 \\ -.0215 & \pm .0708 & 4.0835 \end{pmatrix}$$

Both of the signs on the  $A_{23} = A_{32}$  element must be considered since more than one crystal was used to obtain the data, and it was impossible to distinguish between positive and negative senses of rotation for different crystals. We therefore obtain two different tensors, and additional data will be required to determine which tensor is correct. One method of deciding between these is to observe the rotation dependence when the crystal is mounted with one of the principal axis perpendicular to the field. These experiments are in progress.

Choosing the element  $A_{23}$  positive gives the principal g-values as

$$\lambda_1 = 4.0180$$

$$\lambda_2 = 4.02895$$

$$\lambda_3 = 4.1842$$

where the  $\lambda$ 's are the squares of the principal g values. Diagonalizing, we obtain an orthogonal matrix whose columns are the vectors  $l_{xk}$ ,  $l_{yk}$ ,  $l_{zk}$  where the l's are the direction cosines between the crystallographic axis and the principal axis. This matrix is

$$\begin{pmatrix} .9574 & .15995 & -.2333 \\ .2884 & -.5695 & .7713 \\ .0003 & .8062 & .5922 \end{pmatrix}$$

If we choose the element  $A_{23} = A_{32}$  as negative we obtain the squares of the principal g-values as

$$\lambda_1 = 3.9920$$

$$\lambda_2 = 4.0633$$

$$\lambda_3 = 4.1760$$

In this case, the matrix of the direction cosines becomes

$$\begin{pmatrix} .7173 & .6932 & .0808 \\ .4483 & -.3822 & -.8012 \\ .5334 & -.6114 & .5928 \end{pmatrix}$$

The spectrum  $\text{KH}_2\text{PO}_4$  and  $\text{KD}_2\text{PO}_4$  was recorded from 77°K through about 160°K, the only observable change being that the spectrum disappeared at 160°K. There is also no observable change in the spectrum at varying power levels within the range of the spectrometer.

Since the isotropic hyperfine coupling is the same for the deuterated as for the undeuterated the hyperfine coupling must be to the  $^{31}\text{P}$  nucleus. The coupling for an electron in a 3s orbital on a phosphorus has been found to be 3600 gauss'. Since the observed splitting is 33 gauss this leads to a spin density of  $33/3600 \approx .9\%$  on the phosphorus. The remaining 99.1% of the spin density is believed to be on an oxygen with a broken hydrogen bond. One might expect such a radical to give an esr spectrum with a relatively large anisotropy. The g anisotropy, of course, arises from the "crystal field" (in this case a ligand field). The direction

of one of the g axis lies close to the P-O bonds, as might be expected in this case. It is thus concluded that the free radical is an  $\text{HO}_2\text{PO}\cdot$  radical.

During the writing of this report an abstract appeared in the Bulletin of the American Physical Society<sup>2</sup> describing similar work in electron irradiated  $\text{KH}_2\text{PO}_4$  and  $\text{KD}_2\text{PO}_4$ . (The Bulletin was received April 2, 1963). The results and conclusions of this work for  $\text{KH}_2\text{PO}_4$  agree with those given in the abstract. (The g tensor did not appear in the abstract so no check can be made on this.) However, our work is in disagreement with the work reported in (2) on  $\text{KD}_2\text{PO}_4$ . These investigators find only an isotropic g in the deuterated crystal, the difference in the two spectra being ascribed to a rapid tumbling of the free radical averaging the anisotropy in the g tensor. On the other hand we find no change in the spectrum except for the narrowing of the lines and the appearance of two weak extra lines with the same splitting as the strong lines but with a nearly isotropic g tensor centered near  $g = 2.015$ . These lines may be present in the undeuterated crystal.

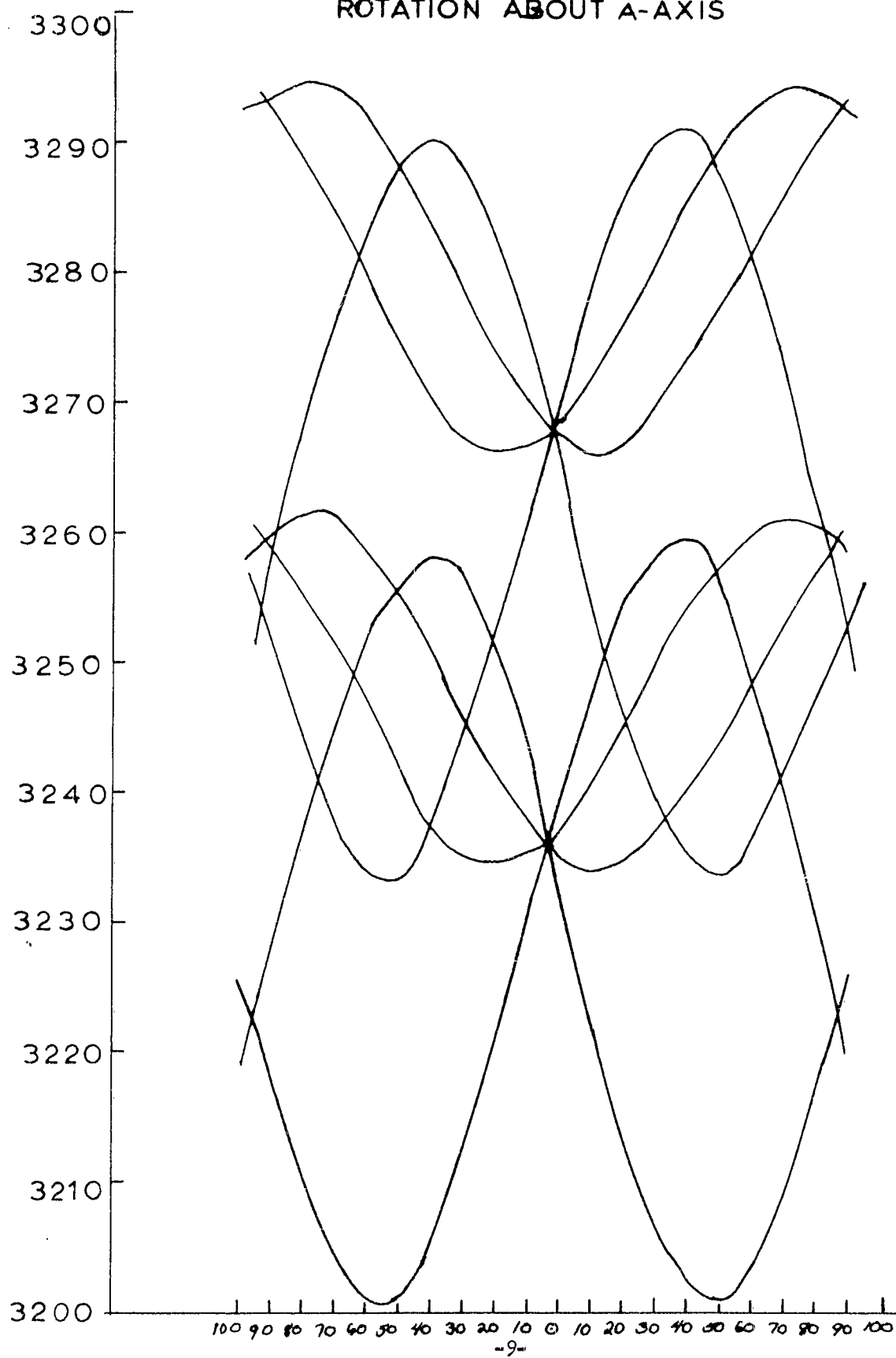
There are two possible reasons for this discrepancy. One is a possibility of a difference in the composition of the deuterated crystals used in the two experiments and the other is that there may be two species of free radicals formed in the deuterated material, one being dominant for x-irradiation, the other for electron irradiation. It is believed that the latter alternative is the more probable. Arrangements are being made to electron irradiate one of our deuterated crystals as soon as possible.

If an isotropic  $g$  is found, then two kinds of centers must be formed, one dominant for x-irradiation and the other for electron irradiation. If this is the case, a check on the postulate that the isotropic  $g$  is due to tumbling will be made by examining the spectrum at 4.2°K where any such motion should be frozen out. This work should be complete within two weeks.

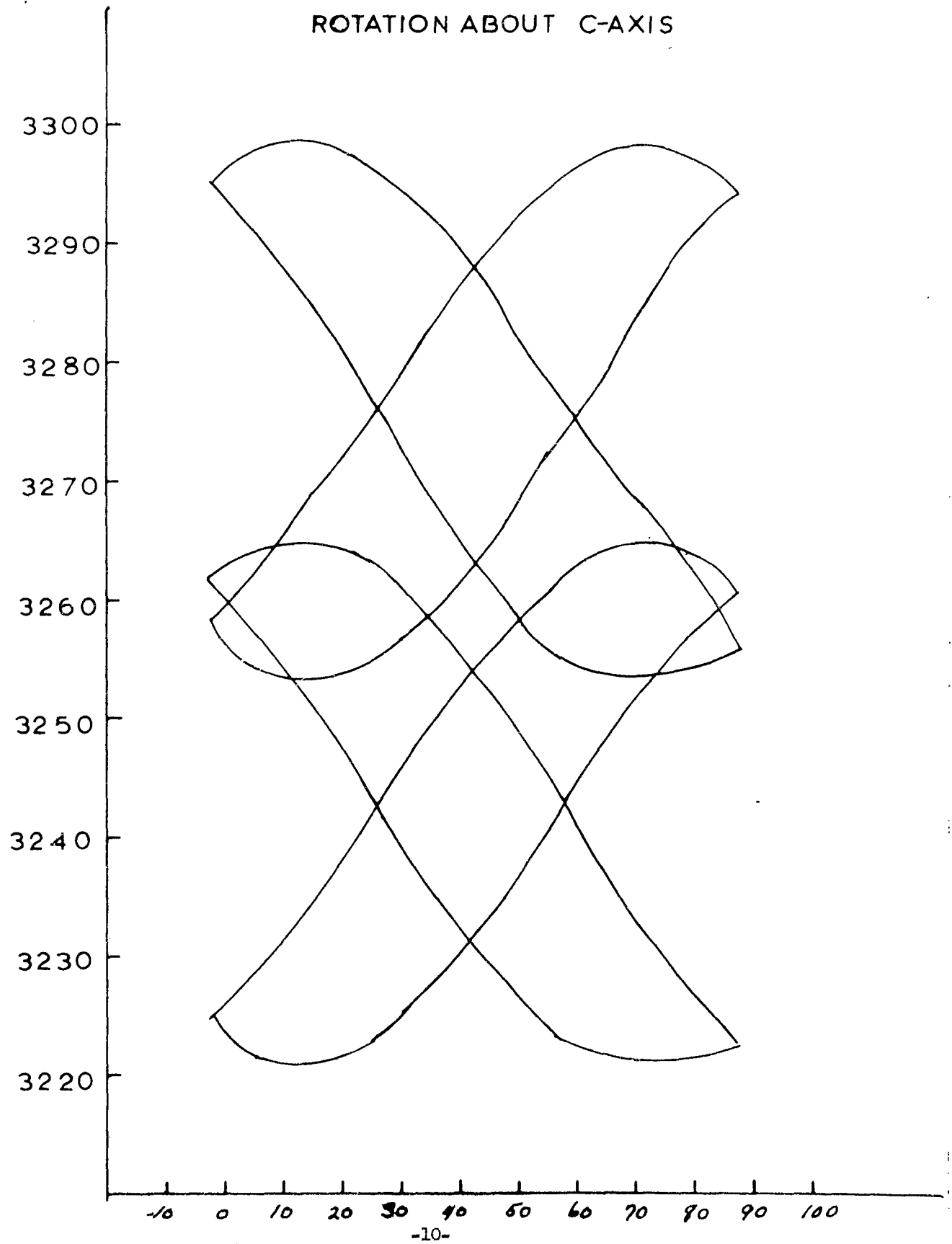
The above work will be basis of the Ph.D. dissertation of Mr. W. E. Hughes.

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# ROTATION ABOUT A-AXIS



# ROTATION ABOUT C-AXIS



## 2. ESR of X-Irradiated Rochelle Salt

An independent program which received partial support under this grant was a study of the kinetics of free radical formation and decay in Rochelle Salt. It has been observed previously that 3 distinct species of paramagnetic centers are produced by x-irradiation of rochelle salt. After x-irradiation, center #1 decays relatively rapidly and monotonically, #2 grows and then decays, while #3 grows slowly and monotonically. The spectrum remains stable after about 2 weeks. The kinetics suggest  $1 \rightarrow 2 \rightarrow 3$ . The stable center has previously been identified as a broken CH bond. A large dose of uv ( $\approx 335$  m $\mu$ ) has been observed to exactly reverse the decay of the centers, approximately in proportion to dose. After uv irradiation, they redecay to the same stable configuration with no measurable deviation in decay rates. uv irradiation of a crystal not previously x-irradiated produced no observable centers. It has also been observed that the uv absorption spectrum changed after x-irradiation, absorption increasing in a band centered at  $\approx 290$  m $\mu$  and decreasing in a band centered at  $\approx 245$  m $\mu$ . The times needed to reach stable configurations correlate well with those for ESR transient effects. A paper describing this work was presented at the St. Louis Meeting of the American Physical Society.<sup>1</sup>

In addition one of the unstable free radicals has been identified. The unpaired spin on the carbon is in a pure p orbital with the remaining three carbon bonds in the nodal plane. This requires a movement of the O-H group into the plane of the carbon atoms. One of the unstable

radicals has been shown to be the same radical before this motion occurs. The hydrogen of the O-H group which must move is hydrogen bonded to a water of hydration and one would expect, therefore, its motion to be hindered. A calculation is in progress to predict the spin densities on the carbon and on the hydrogen on the adjacent carbon atom theoretically.

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1. Gamble, Moulton and Moulton, Bull. Am. Phys. Soc. Ser. II, 8, 353 (1963).



### 3. Frequency Shifts of $\text{Cl}^{35}$ Pure Quadrupole Resonance in $\text{NaClO}_3$ at Low Temperatures

#### (a) Introduction

The temperature dependence of pure quadrupole resonance frequencies have been studied in several compounds previously.<sup>1-4</sup> The theory of the shifts has been developed by Bayer<sup>5</sup> and modified by Kushida.<sup>6</sup> None of the above measurements were extended below 15°K, and in most cases only a few points between room temperature and liquid air temperature are available. The most complete study was made by Vanier,<sup>3</sup> who has obtained complete data from 77°K to 15°K, and found excellent agreement with the theory Bayer and Kushida. In all cases the value of  $\nu(0)$ , the frequency at absolute zero, has been obtained by some means of extrapolation. Since there has been a paucity of work done at low temperatures, it was felt that more complete data, especially between 4.2°K and 20°K, would provide a better check on the Bayer theory, and would give better determination of the moment of inertia and rocking frequencies of the  $\text{ClO}_3$  group.

The experiments indicate that while the gross features of the temperature dependence are described by the Bayer theory, complete agreement was not found, since least square fits of the data to the Bayer theory give somewhat different values of the parameters for fitting over different temperature ranges. If one considers the approximations and assumptions inherent in the Bayer theory, one would expect the low-temperature region to give the best agreement.

(b) Instrumentation

The measurements were made with a Pound-Knight-Watkins type spectrometer modified to permit frequency modulation. The circuit diagram is shown in Fig. 1. Standard narrow band amplifier and phase sensitive detection methods were used. The output of the spectrometer was coupled to a Hewlett-Packard frequency counter. The frequency of spectrometer was swept slowly through the resonance and at the line center, as determined by observing the zero of the observed line derivative on the chart recorder, the spectrometer frequency was counted. It is felt that the frequency measurements are accurate to  $\approx \pm 200$  c/s. The temperatures were measured with a Minneapolis-Honeywell germanium resistance thermometer (Series II) calibrated from 4.2°K to 100°K. A Leeds and Northrop potentiometer type K-2 was used to measure the resistance. The temperatures are accurate to within the calibration of the thermometer,  $\pm 0.1^\circ\text{K}$ . The thermometer was imbedded directly in the sample.

The sample coil was connected to the spectrometer through a stainless steel coaxial line. The sample was thermally connected to the helium bath by means of a copper cup containing the sample and a 1/8" brass rod extending into the helium bath. A 1/2 watt 50 ohm carbon resistor was attached with epoxy resin to the outside of the copper cup so that by varying the power in the resistor a temperature range from 4.2°K to 50°K could be covered. It was found that with a given power input the time rate of change, after stabilization, of the temperature did not exceed .1° per minute. This small rate of change of the temperature plus the fact that no line broadening occurred indicates that temperature gradients in the sample were negligible.

The samples of  $\text{NaClO}_3$  used were powder samples of "Bakers Analyzed Reagent Grade" directly as supplied by the manufacturers. It was found necessary, to avoid saturation in the low temperature region, to give the sample a dose of 700 kilo roentgens of 2 mev  $\gamma$  radiation. In view of the results of Benedek and Kushida<sup>7</sup> concerning the effect of the addition impurities on the resonance frequencies, it is felt that the effect of radiation on the quadrupole frequency is completely negligible.

(c) Results and Discussion

The energy levels of a quadrupole system in an axial symmetric field gradient is given by

$$E_m = \frac{eQq_{zz}}{4I(2I - 1)} \left[ 3m^2 - I(I + 1) \right]$$

which gives

$$h\nu_0 = \frac{eQq_{zz}}{2}$$

for

$$I = \frac{3}{2}, \quad m = \pm \frac{3}{2}, \quad \pm \frac{1}{2},$$

where  $Q$  is the nuclear quadrupole moment,  $q_{zz}$  is the field gradient at the site of the nucleus,  $I$  is the nuclear spin quantum number, and  $m$  is the nuclear spin projection quantum number. Bayer has shown that if the field gradient  $q_{zz}$  is modulated by a harmonic torsional oscillation of

the lattice, and if the energy of this oscillation is equated to the mean energy of a system of Plank oscillators, that the frequency is given by

$$\nu(T) = \nu_0 \left( 1 - \frac{3}{2} \frac{h}{4\pi^2 \textcircled{+1} \nu_L} \right) \left( \frac{1}{2} + \frac{1}{e^{\frac{h\nu_L}{kT}} - 1} \right) \quad (1)$$

where  $\nu_L$  is the frequency of the lattice vibration, and  $\textcircled{+1}$  is the moment of inertia of the rigid lattice; and  $\nu_0$  is the above expression. The lattice vibrations correspond to a tilting motion frequency must be much greater than the Larmor frequency of the nucleus. Kushida has pointed out that in general there are more than one modes which contribute to this effect; in  $\text{NaClO}_3$  these are the two tilting motions of the  $\text{ClO}_3$  group about two orthogonal axes as shown in Fig. 2. Neglecting intermolecular coupling, these are approximately normal modes; according to Kushida, the temperature dependence in such a case will be given by

$$\nu(T) = \nu_0 \left\{ 1 - \frac{3h}{8\pi^2} \sum_i \frac{1}{\textcircled{-} i \nu_i} \left( \frac{1}{2} + \frac{1}{e^{\frac{h\nu_i}{kT}} - 1} \right) \right\} \quad (2)$$

where the summation includes the two aforementioned modes. Assuming that the two modes are approximately equivalent in this case, the expression becomes

$$\nu(T) = \nu_0 \left\{ 1 - \frac{3h}{4\pi^2 \textcircled{-} \nu_L} \left( \frac{1}{2} + \frac{1}{e^{\frac{h\nu_L}{kT}} - 1} \right) \right\}$$

which may be written as:

$$\nu(T) = \nu(0) - \frac{A}{e^{\frac{\lambda}{T}} - 1}$$

where

$$\nu(0) = \nu_0 \left( 1 - \frac{3h}{8\pi^2 \Theta \nu_e} \right)$$

is the frequency corresponding to the ground-state amplitudes of the lattice vibrations, and

$$A = \frac{3h\nu_0}{4\pi^2 \Theta \nu_e}$$

$$\lambda = \frac{h\nu_e}{kT}$$

The data were fitted to the above equation by a least-square fit on a Remington Rand SS80 computer. Three ranges of the data were fitted to this equation. Number one consisted of all data points taken in this laboratory. There were 48 of these, 15 of which were at approximately between temperature, thus weighing the lower temperature region more heavily. Number two included only the points above 16°K. Number three included all data points plus the value of the frequency at 78°K measured by Wang. Three sets of parameters  $\nu(0)$ ,  $A$ , and  $\lambda$  were obtained as follows

1.  $\lambda = 131.25^\circ\text{K}$   
 $\nu(0) = 30.68565 \text{ MC}$   
 $A = 196.14 \text{ KC}$
2.  $\lambda = 139.77^\circ\text{K}$   
 $\nu(0) = 30.68 < 36 \text{ MC}$   
 $A = 232.28 \text{ KC}$
3.  $\lambda = 156.16^\circ\text{K}$   
 $\nu(0) = 30.68551 \text{ MC}$   
 $A = 350.92 \text{ KC}$

These may be compared with the value of  $\lambda$  corresponding to the  $103 \text{ cm}^{-1}$  Raman line chosen by Kushida as the one best agreeing with the theory in the linear (high temperature) region. This value is  $148^\circ\text{K}$ . Kushida also computed the moments of inertia,  $(+1)$  of the  $\text{ClO}_3$  ion from crystal dimensions. The values of  $(+1)$  obtained from (1), (2), and (3) are  $288 (10)^{-47}$ ,  $229 (10)^{-47}$ , and  $135 (10)^{-47} \text{ KGM}^2$  as compared with the values  $83.39 (10)^{-47}$  to  $88.59 (10)^{-47}$  of Kushida, depending upon his choice of the lattice parameter  $r_1$ . (Fig. 2.)

It should be noted that it is not possible to fit our data accurately over the entire temperature range using the Bayer-Kushida theory, and in addition the best fits yield different values of the parameters  $A$ , and  $\nu(0)$ , and  $\lambda$ . The fit over a wider temperature could undoubtedly be obtained by treating the modes as non-equivalent and using two terms in the summation of (2). However, from the geometry of the  $\text{ClO}_3$  group this does

not appear justified, since the moments of inertia about the two axis are very nearly the same. Furthermore this would imply the existence of a second Raman line very near the one assumed to be due to the rocking motion producing the quadrupole frequency shift.

One possible explanation of the difficulty in fitting the data over the entire range from  $4.2^{\circ}$  to  $77^{\circ}\text{K}$  is the effect of the contraction of the crystalline lattice. Unfortunately we have been unable to find data on the volume coefficient of expansion of  $\text{NaClO}_3$  in the literature. A more likely explanation seem to us to be that the assumptions and approximations of the Bayer theory are rigorously valid only at very low temperatures. It is our opinion that the values obtained for the fitting parameters over the range of our data ( $4.2^{\circ}$  to  $54^{\circ}$ ) are the best values.

A manuscript based on this work is in preparation and will be submitted to the Journal of Chemical Physics. It forms the basis of the M.S. thesis of Mr. W. D. Partlow.

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  2. T. C. Wang, Phys. Rev. 99, 566 (1955).
  3. J. Vanier, Canadian J. Phys. 38, 1937 (1960).
  4. T. Kushida, G. B. Benedek, N. Bloembergen, Phys. Rev. 104, 1364.
  5. H. Bayer, Z. Physik 129, 401 (1951).
  6. T. Kushida, J. Sci. Hiroshima Univ. A, 19, 327 (1955).
  7. G. B. Benedek and T. Kushida, Rev. Sci. Inst. 28, 92 (1957).

# R.F. SECTION OF FREQUENCY MODULATED SPECTROMETER

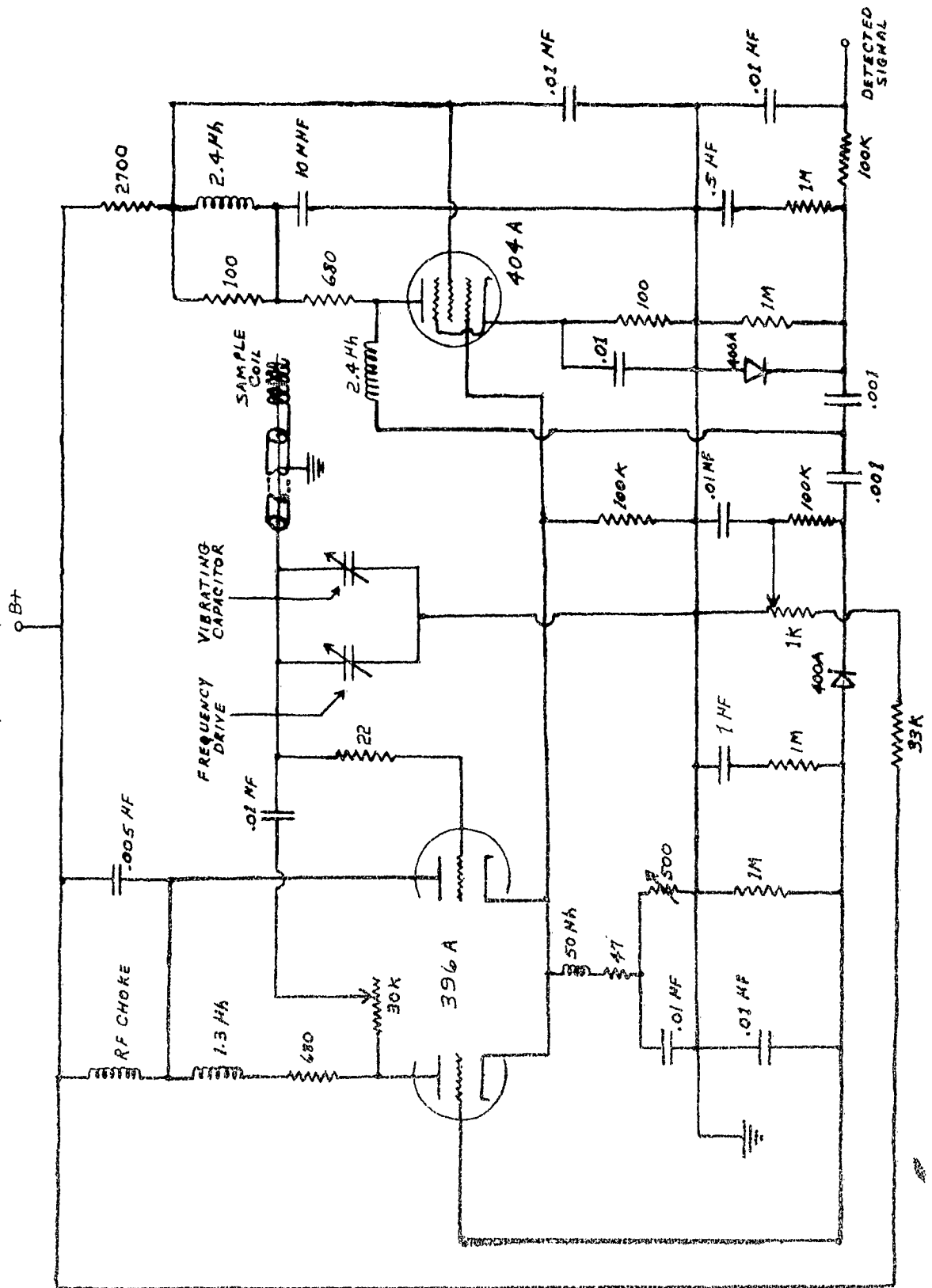
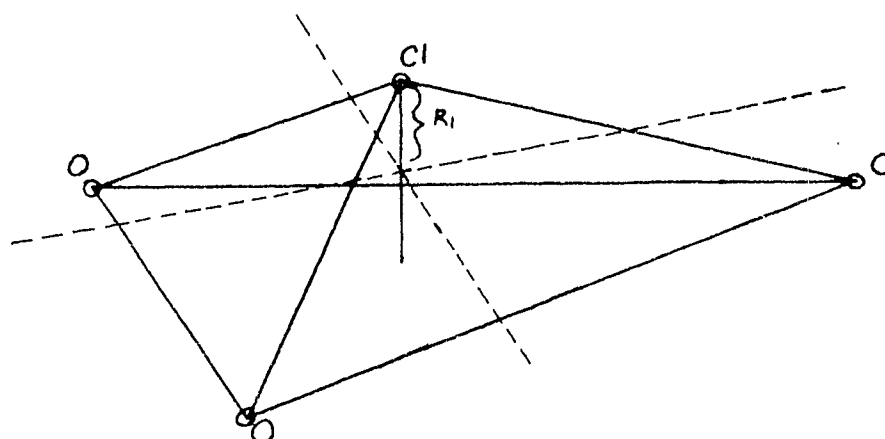


Fig 1.

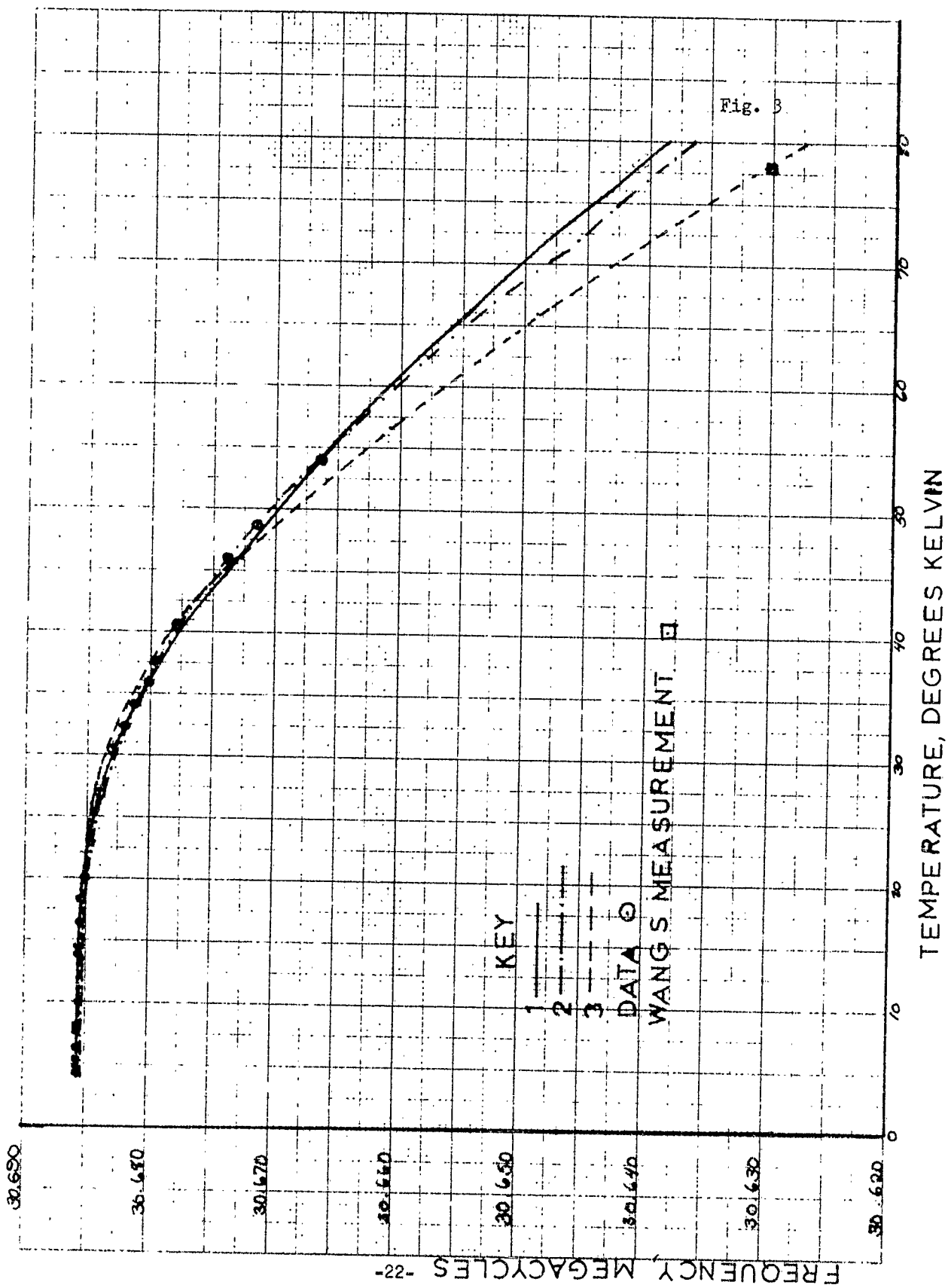


FIG. 2



MUTUALLY PERPENDICULAR AXES OF  
TORSIONAL VIBRATION (DASHED LINES) OF  
THE  $\text{ClO}_3$  PYRAMID, (AFTER KUSHIDA).

Fig. 3 20 X 20 THE INCH 350-101-G



#### 4. Spin-Lattice Relaxation of $^{79}\text{Br}$ and $^{81}\text{Br}$ in $\text{NaBrO}_3$

Another research area in pure quadrupole spectroscopy being carried out (this problem has been only partially supported by this grant) is an investigation of the spin-lattice relaxation times and frequency shifts in  $^{79}\text{Br}$  and  $^{81}\text{Br}$  isotopes in  $\text{NaBrO}_3$  at low temperatures. Due to the presence of the two isotopes with different electric quadrupole moments and magnetic dipole moments it has been possible to separate different relaxation mechanisms and correlate the spin-lattice relaxation with the frequency shifts. Data on both isotopes from 4.2°K to 16°K have just been completed, and it is anticipated a manuscript describing this phase of the work will be submitted for publication within the next few months. Many more potentially fruitful experiments will be carried out in this area as soon as this phase of the work is completed. Examples of such experiments will include relaxation measurements of individual zeeman components to separate out the various relaxation rates, spin mixing experiments, etc. A preliminary report of the initial phase of this work was presented at a meeting of the Southeastern Section of the American Physical Society (Bull. Am. Phys. Soc. Ser. II 7, 574 (1962)). The new work will be completely sponsored by AFOSR under the new grant.

Publications Under This Grant

1. R. F. Tipsword and W. G. Moulton, Bull. Am. Phys. Soc. Ser. II, 7, 574 (1962).
2. W. L. Gamble, Grace C. Moulton and W. G. Moulton, Bull. Am. Phys. Soc. Ser. II, 353 (1963).